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14. ABSTRACT Electro-spark alloyed (ESA) surfaces were developed as an erosion barrier for Ti-6-4 impellers on shaft driven compressors (SDCs). Coatings based on tungsten carbide, tantalum carbide, titanium carbide all with a cobalt matrix were evaluated for high velocity particle erosion in conventional wear test studies as well as wind tunnel testing. In both cases, the coating survived erosion by alumina and silica sand ranging in size from 50 to 200 microns. The ESA process formed these coatings with a full metallurgical bond, a nano-grained texture, and no heat affected zone in the base titanium alloy. The coatings demonstrated life extensions ranging from 3 to over 20 times, based upon test conditions. The ESA process was also demonstrated on actual impeller hardware for manufacturing feasibility considerations. This was a Phase I SBIR through NAVAIR, Pax River, MD.					
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Navy Phase I SBIR Report: Topic Number N00-002

**Phase I
FINAL REPORT**

Compressor Impeller Erosion Resistant Surface Treatment

**Contract No.: N68335-00-C-0336
Start Date: 3 April 2000**

**Prepared for:
Naval Air Warfare Center – Aircraft Division
ATTN: Dr. George Richardson, Code 4.3.4.2
48066 Shaw Road
Bldg 2188, Room 115
Patuxent River, MD 20670**

**Prepared by:
Key Personnel: Michael A. Riley
Surface Treatment Technologies, Inc.
1110 Business Parkway, South
Westminster, MD 21157**

Declaration of Technical Data Conformity (JAN 1997).

The Contractor, Surface Treatment Technologies, Inc. hereby declares that, to the best of its knowledge and belief, the technical data delivered herewith under Contract No. N68335-00-C-0336 is complete, accurate, and complies with all requirements of the contract.

Date: 9 October 2000

Name and Title of Authorized Official: Michael A. Riley, President

Patents – Reporting of Subject Inventions.

No inventions have occurred under this Phase I SBIR contract.

Final Scientific or Technical Report.

The Contractor shall submit two copies of the approved scientific or technical report delivered under this contract to the Defense technical Information Center (DTIC), Attn: DTIC-OC, 8725 John J. Kingman Road, Suite 0944, Fort Belvoir, VA 22060-6218. The Contractor shall include a completed Standard Form 298, Report Documentation Page, with each copy of the report.

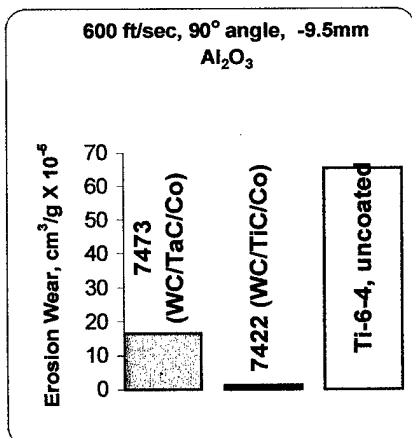
Executive Summary

Surface Treatment Technologies, Inc, ST2, has developed advanced ceramic composite coatings for titanium alloys employed on the V-22 aircraft for shaft driven compressors. In this Phase I SBIR effort, the following key goals have been met::

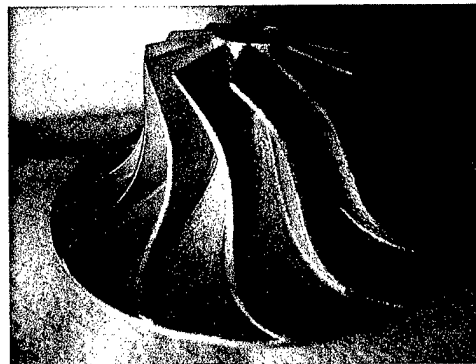
- the electro-spark alloying (ESA) process have identified two different ceramic chemistries that form compatible coatings with Ti-6-4
 - tungsten carbide/tantalum carbide/cobalt
 - tungsten carbide/titanium carbide/cobalt
- the coatings developed offer the following structural benefits
 - full metallurgical bond between coating and substrate
 - nano-grained ceramic composite coating
 - no heat affected zone in the titanium base alloy
 - the technology has been demonstrated on the impeller hardware
- wear testing has shown that the coatings survive and extend the life of Ti-6-4
 - in-house ASTM erosion tests with alumina grit
 - independent wind tunnel erosion tests conducted by the University of Cincinnati

The ST2 team has provided surface coatings that have outperformed the base titanium alloy from 6 to 60X under a wide variety of erosion media type, size, and wind velocity. In this report, coating procedures have been performed on impeller hardware, and have also been conceptually designed for robotic automation in manufacturing scale-up.

St2 has also provided concise recommendations to move forward in both the Phase I Option and the Phase II SBIR. These recommendations take the process from its current coupon development stage through full-scale pilot production using robotic tooling with specific hardware algorithm development.



Wear test comparative data



Electro-Spark coated impeller hardware

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Introduction

The Armed Services presently use shaft driven compressors (SDC's) on certain aircraft to provide, among other things, service to on-board inert/oxygen gas separators (OBIGS/OBOGS) and environmental control systems (ECS). Air intakes for these compressors are equipped with particle separators to prevent abrasive material from contacting the impeller. The impellers can operate from 87,000 to 100,000 rpm's (nominal) and at temperatures from 125_F (degrees F) to 600_F (degrees F). Aircraft, particularly helicopters and other vertical/short take-off and landing (VTOL/STOL) aircraft such as the V-22 Osprey, when operating over sandy or dusty landing zones (LZ's) or during dust/sand storms, have experienced rapid erosion of impellers, especially when the particle separator is overtaxed. This can lead to loss of function of critical components and potentially catastrophic system failure. There is a need to provide a surface treatment for SDC impellers, currently made of titanium 6-4 alloy, which eliminates the erosion phenomena or obviates it to an acceptable level. Recent data collected by the Navy on such impellers has shown significant loss of material, with as much as 0.5 inches removed from the impeller leading edges in a little over 120 hours of flight time. A photo of the V-22 impeller in question before and after particle erosion is provided in Figure 1.

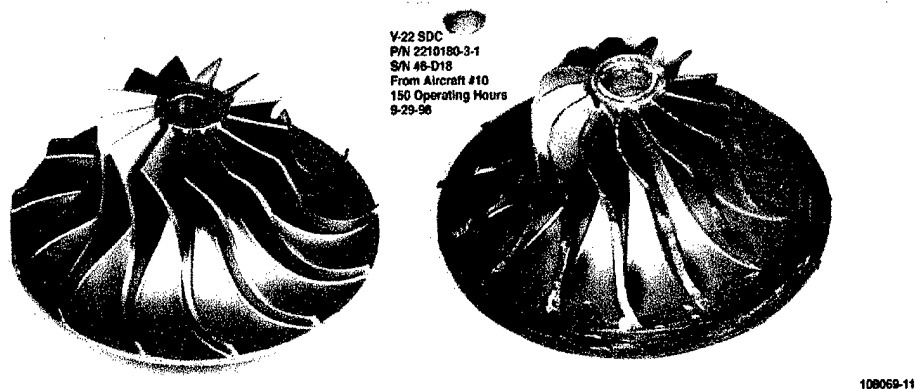


Figure 1. V-22 SDC impeller as new (left) and after 150 hours of flight time (right). *Photo courtesy of NAVAIR*

While the need in this solicitation focuses on the particular problem faced by the V-22 Osprey, the greater need faces many other aircraft, both fixed wing and rotary. Moreover, similar hardware in power trains for ground vehicles have also undergone severe erosion in desert environments based on data collected during Desert Storm.

The problem is exacerbated by both the need for extremely high rotational speeds and the need for lightweight materials such as titanium 6-4. Ti-6-4 is a high strength alloy at temperature, with very low density, but is not good in wear and erosion conditions. This application is particularly difficult to correct since the blade tip velocities are very high, and the particles impacting the blades are a mixture of silicon and aluminum oxide sand, approximately 200 grit in size.

An additional difficulty posed by this problem is the current state of the art in coating technologies. Most high wear surfaces available today are attached to the substrate metal by mechanical adhesion alone. The best of these technologies, High Velocity Oxy-Fuel deposition, or HVOF, can offer a near-fully dense surface with an adhesion strength of ~ 20 ksi. While this is good for thermally sprayed coatings, it is insufficient for such an erosive environment. Recent studies on other hardware exposed to the same desert environment show the inability of HVOF to meet such demands. Currently, the U.S. Army Hellfire missile is undergoing a retrofit due to sand erosion of a missile latch release mechanism. Vibrations in the system result in high frequency impacts between the missile body and the latch, which is composed of high hardness 17-4 PH steel. HVOF attempts to coat this latch with tungsten carbide have failed under tests involving wear/erosion with 200 grit aluminum oxide, with no high velocity impact component involved in the wear/erosion event, but rather simple mechanical abrasion. In these tests, the coating simply cracked and abraded from the substrate metal.

The active mechanisms in the current V-22 impeller problem are most likely strictly high velocity impact erosion. Based on data gathered from the NAVAIR customer, the edges of the impeller pictured in Figure 1 are uniformly worn from the outermost edges toward the center. As much as 1/2 inch of material has been removed from the thinnest sections at the top, but smoothing/rounding has occurred over the entire edge of each blade. While it is clear that a part of the solution must lie in better filtration. It is also important to protect the blades as much as possible when particles overwhelm the filtration system.

For a coating to be successful, it must do the following:

- provide sufficient hardness to withstand the impact erosion forces of high velocity particle impingement
- form a true metallurgical bond with the substrate alloy to prevent mechanical erosion
- offer a crystalline structure that promotes both wear resistance and lubrication to minimize the impact forces
- create no thermal stresses in the impeller, nor weaken the structure through the creation of a heat-affected-zone (HAZ)
- be a highly reproducible process (meet ISO and MILSPEC requirements)
- be affordable

Based upon developments to date, titanium may eventually be replaced, yet a coating for the impeller system may still be a requirement due to the harsh operational environment. Based on the critical operational criteria set forth in this customer need, many conventional approaches can already be dismissed based on available data. As discussed above, coatings that rely on mechanical attachment offer insufficient strength to survive the environment. Many coatings that offer metallurgical bonding must be applied through a heat treatment process. This has the potential of severely degrading the strength of the titanium impeller, or at least causing the development of a new heat treatment cycle for the hardware. Moreover, a bulk-clad surface, like a Colmonoy braze, a Duraclad, or a Conformaclad, will increase the wall thickness to the point where impeller redesign may be necessary to obtain the required power requirements from the unit. The pure ceramic solution, such as silicon nitride may now be available due to breakthroughs in nano-powder processing, but this will entail considerable expense over current costs, and such systems will always have the potential for undergoing catastrophic failure, rather than a gradual erosive failure.

Background: Electro-Spark Alloying

Surface Treatment Technologies, Inc. (ST2) has studied the impeller problem extensively, and offers the following surface treatment as a potential solution. The technology known as electro-spark alloying (ESA) has evolved over the past ten years in a research environment, and may offer the type of wear coating required by this application. ESA is a micro-welding process in which an electrode is applied to a surface with a pulsed current mode. In addition, the electrode either rotates or oscillates as it is in contact with the base metal being coated, as shown in Figure 2. During this process, minute quantities of the electrode material are transferred through the arc and deposited on the substrate metal. This approach transforms the electrode material, which can be made from any electrically conductive metal or ceramic, and made of any grain size, into an amorphous or nano-crystalline coating that is metallurgically bound to the substrate metal. A coating is then developed by rastering the electrode over the area to be coated, and slowly building up the coating layer. ESA is a relatively slow process, forming a coating of 1-3 mils in thickness at a rate of ~ 2-3 square feet per hour. However, for applications like the one in this solicitation and other small parts requiring wear/erosion or corrosion protection, ESA is an excellent approach.

As shown in Figures 3 and 4, ESA can be adapted to full automation or applied simply by holding the electrode in hand and rubbing it back and forth over the area to be coated. A key aspect of this process is the type of material formed by the deposition process. Depending on the conductivity and melt/vaporization temperature of the electrode, the coating can be either amorphous or nano-

crystalline in nature. This is a major coatings breakthrough, since much emphasis has been placed on the development of nano-crystalline materials as a

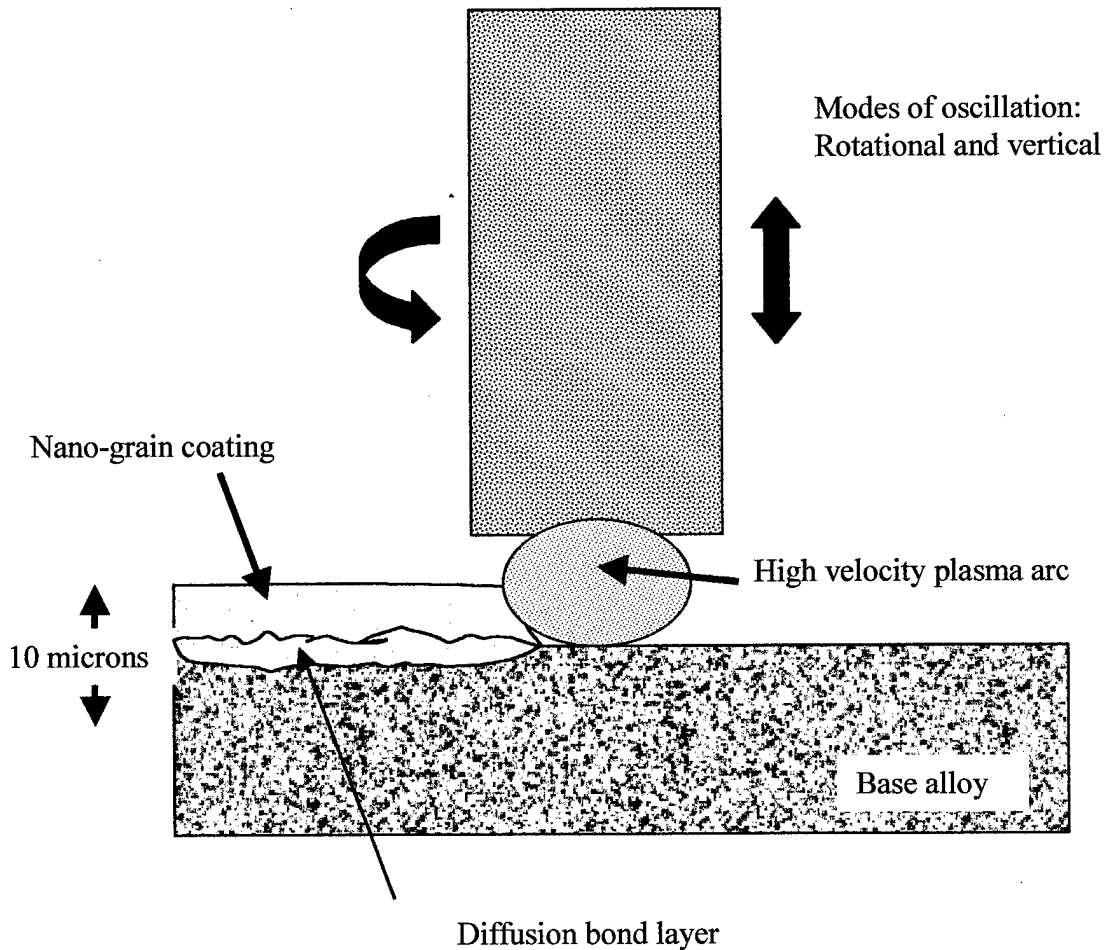


Figure 2. Schematic of the ESA process, showing the electrode transfer into the bulk alloy.

powder for coating applications. In the ESA process, no nano-powders expense is required, since an electrode of conventional grain-sized alloy, can be transformed into a nano-grained surface by virtue of the arc process. A micrograph of such a surface is shown in Figure 5, and wear data from a nano-grained surface is compared directly with the wear data from a coating of the same chemical composition, but with a larger grain size (Figure 6). Another benefit of the combined nano-grained structure and metallurgical bonding is the ability of the coating to remain intact, even under sever bend conditions, as shown in Figure 7, where ESA tungsten carbide is compared directly to detonation-gun tungsten carbide. One more key benefit of the ESA process lies in its ability to form wear and lubricious coatings using a multi-layered approach. Figure 8 shows two ESA coatings, known as wear-tech and slip-tech, both of

Surface Treatment Technologies, Inc.

which have coefficients of friction that approach that of Teflon, yet are full-metal and metal/ceramic layers.

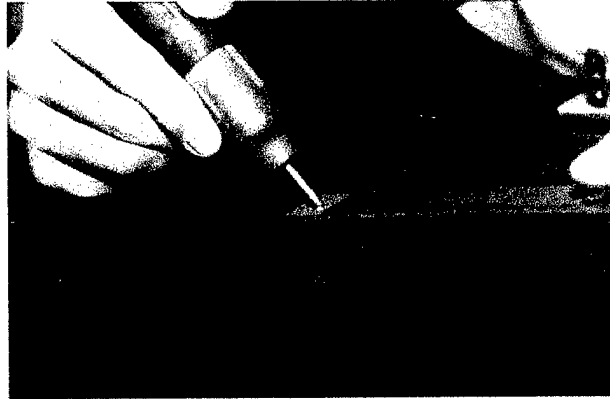


Figure 3. Hand operated ESA system

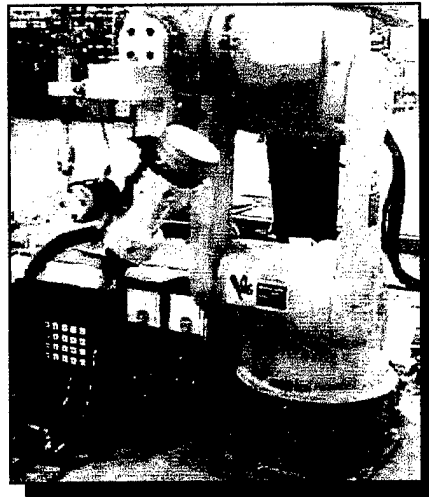


Figure 4. Multi-axis controller unit operating ESA in a production facility

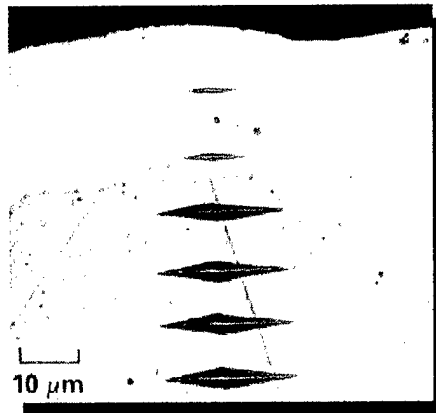


Figure 5. Micrograph of ESA chromium carbide on 316 stainless steel, showing micro hardness indentations. Note the lack of a discernable heat affected zone from the coating to the base alloy.

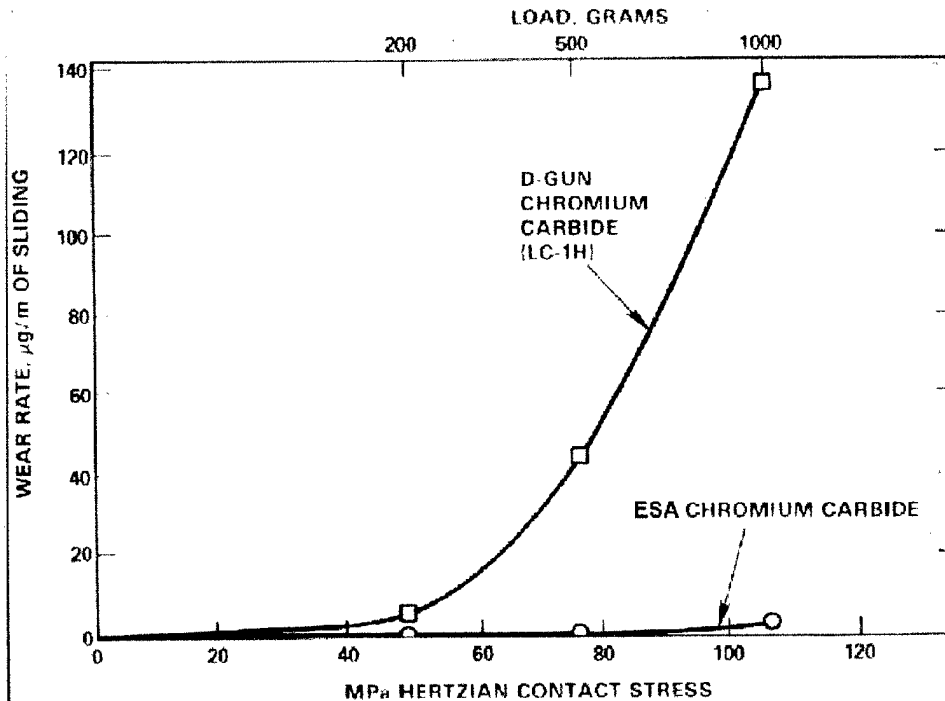


Figure 6. Wear data comparing chromium carbide (on stainless steel) coatings produced by ESA vs. detonation gun.

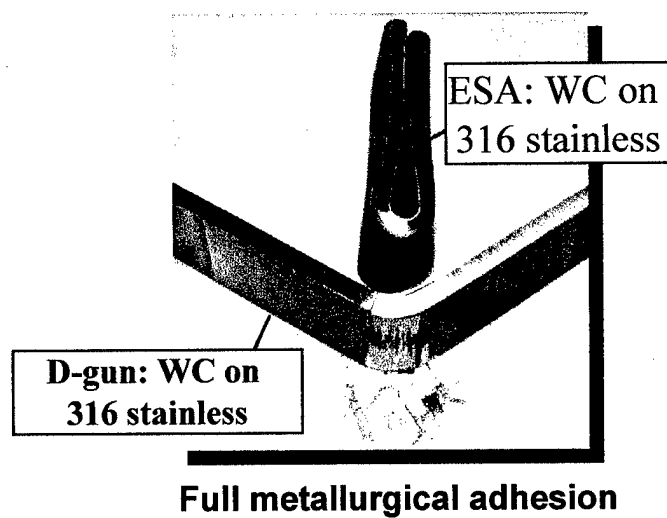


Figure 7. ESA adhesion comparison of tungsten carbide vs. that of detonation gun coatings.

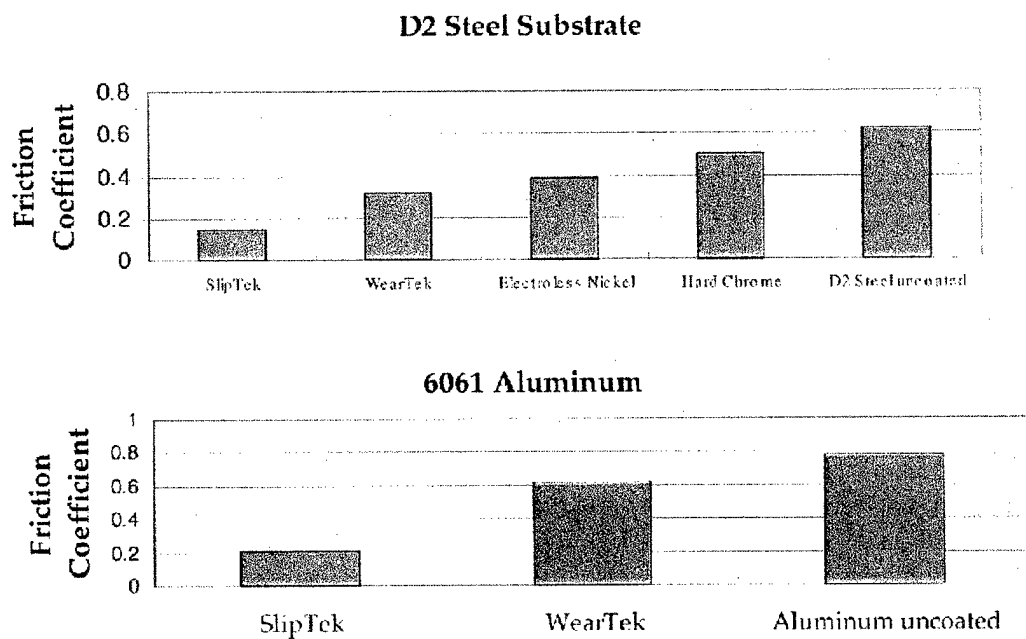


Figure 8. Coefficient of friction of ESA coatings "Wear-tech" and "Slip-tech".

ESA has been applied in recent research efforts to a wide range of materials and end uses, and the coatings and markets of interest are identified in Table 1. Finally, in a specific example of wear enhancement for titanium 6-4- alloys, Figure 9 shows carbide and carbide/molybdenum coatings as wear reduction enhancements against sliding friction wear. Here, the potential for an ESA solution on titanium alloys is clearly demonstrated. All of these case studies are based on layers that are not more than 5 mils thick, but that often employ a multi-layered structure.

From this brief introduction of the proposed solution, it is clear that ESA processing offers a new, innovative way of forming wear/erosion coatings with nano-grained structure. The process is far more affordable than evolving nano-powder approaches, can be performed in open air with a very small gas-shroud in the vicinity of the discharge, and can be readily automated with commercial-off-the-shelf (COTS) robotics. Moreover, partnership agreements with the ESA hardware developers, Advanced Surfaces and Processes, Inc., Portland, OR, permit ST2 to access ESA for a wide range of market applications. ST2, based on its depth of experience in metallurgically bound coatings, is uniquely qualified to develop the ESA process for military applications, as well as a myriad of commercial applications.

▪ **Wear Resistance**

- Hard Carbides (of W, Ti, Cr, Ta, Mo, Hf, Zr, Nb, V)
- Hardfacing Alloys (Stellites, Triballoys, Colmonoys, etc.)
- Borides (of Ti, Zr, Ta)
- Intermetallics and Cermets

▪ **Corrosion Resistance**

- Stainless Steels
- Special Alloys (Hastelloys, Inconels, etc.)
- Fe, Ni, and Ti Aluminides
- FeCrAlY, NiCrAlY, CoCrAlY

▪ **For Build-up or Special Surface Modifications**

- Ni-base and Co-base Super Alloys
- Nobel Metals (Au, Ag, Pt, Pd, Rh)
- Refractory Metals and Alloys (W, Mo, Ta, Re, Nb, Hf)
- Other Alloys (Fe, Ni, Cr, Co, Al, Ti, Cu, Zr, Zn, V, Sn, Er)

Table 1. ESA coatings investigated to date.

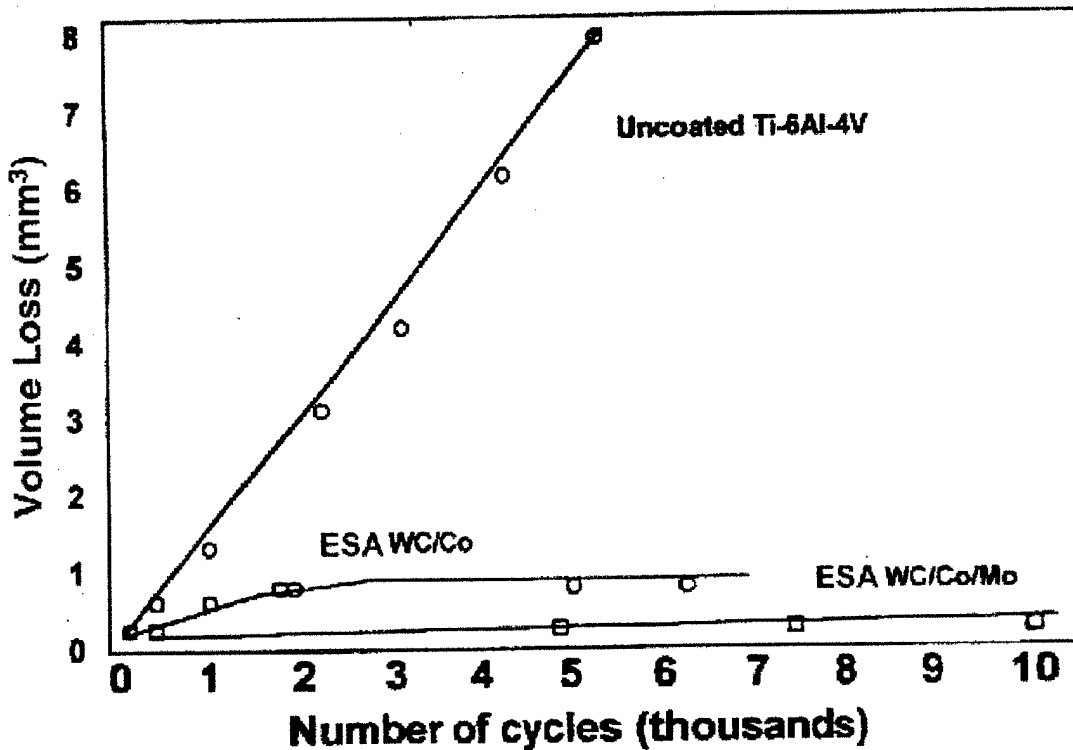


Figure 9. Sliding friction wear comparison of base Ti-6-4, and ESA tungsten carbide/cobalt and carbide/cobalt/molybdenum coated Ti-6-4. Note that the wear essentially becomes a flat-line out to ten thousand cycles.

Phase I Tasks

The following specific tasks were developed in an attempt to address the specific needs of the V-22 impeller program. Note that Task 3, erosion testing was carried out both in-house, as a screening tool, but also by an independent analysis laboratory selected by NAVAIR. Specifically, the University of Cincinnati Mechanical Engineering Department facility was selected due to their highly unique wind-tunnel erosion facility, a facility already used by NAVAIR for erosion testing of rotary wing aircraft components. This work was carried out exclusively under the direction of Professor Widen Tabakof, and had no input from ST2. This point will become a very critical discussion point in the Discussion section of this report.

micro-hardness, and heat affected zone (if any). Optimized parameters will then be used to produce individual coupons for erosion testing. Additional flat test coupons will be produced for small-scale fatigue studies to insure that no additional crack susceptibility has been introduced into the Ti-6-4 as a result of ESA processing. These coupons will be tested to determine the onset of fracture only, not the crack growth rates, as such rates are meaningless in such a high-load component.

Duration: 10 weeks

Personnel: ST2, Advanced Surfaces and Processes

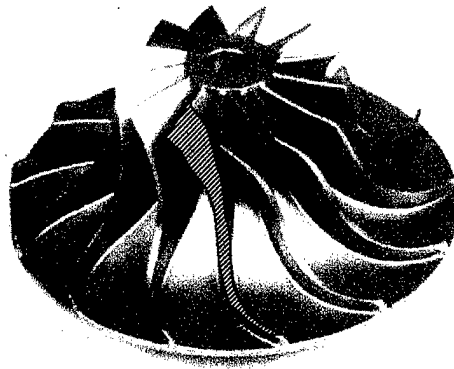
Deliverables: optimized parameters

Task 3. Erosion Testing

ST2 will perform G76-95 or similar test procedures on down-selected alloy surfaces. These tests involve high velocity carrier gas entrainment of aluminum oxide and/or silicon dioxide powders on the test coupon. Weight loss is determined by precise measurement over given time periods. As a follow-up to this test, coupons will also undergo metallographic analysis to determine the efficacy of the coating, or to assess failure mechanisms. If identified under Task 1, optional wear/erosion tests may be conducted, potentially at manufacturer test facilities (in-house or recommended consultant). The outcome of this test sequence will determine the adequacy of selected ESA surfaces to enhance the impeller surface.

Task 4. Manufacturing Feasibility Assessment

Given the complex nature of the impeller in question, ST2 intends to work with the manufacturer to identify potential methods of ESA implementation for scale-up of a production work cell that meets the technical and economic goals of the customer. As shown in Figure 10, the areas marked off in red were the major erosion portions of the impeller in customer conducted V-22 erosion tests. For example, given the flexibility of the ESA process, it may be possible to employ multi-axis robotics to selectively coat portions of the impeller blades, rather than the entire component. Comparisons like this will be conducted to focus on a "narrowed-down" list of manufacturing concepts that will then be further refined and developed into a pilot production cell in Phase II.



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Figure 10. Typical wear areas on V-22 impeller blades.

Duration: 6 weeks

Personnel: ST2, in conjunction with Allied Signal

Deliverables: Manufacturing Feasibility Assessment Report

Task 5. Documentation.

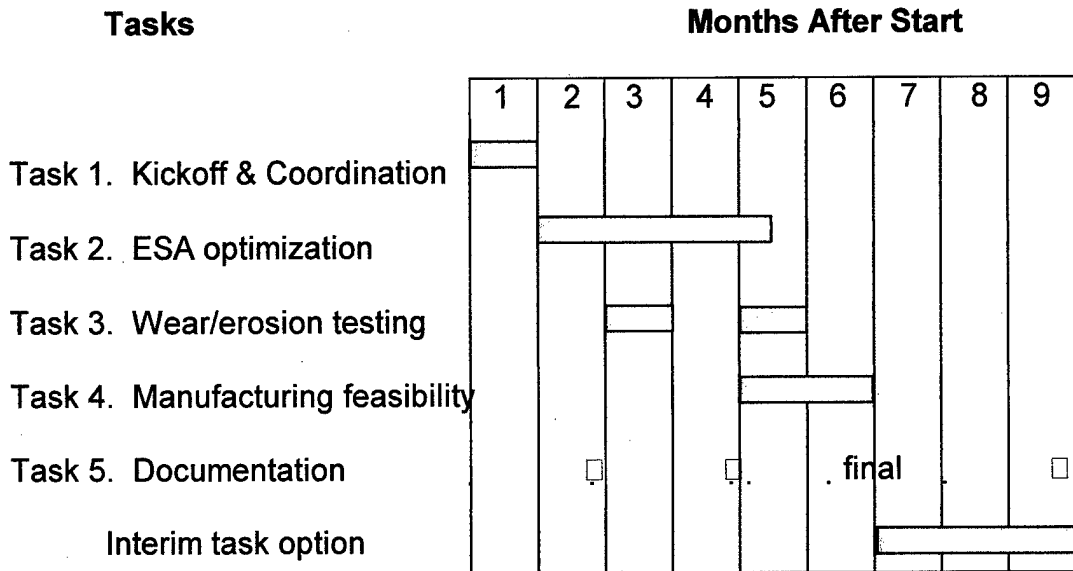
ST2 will document the effort through monthly letter reports, through the deliverables called out in Tasks 1-4, and through a comprehensive final report.

Optional Task.

Given a successful Faze I effort, the Navy permits a bridge funding option between a Phase I and a Phase II SBIR. For this additional 3 months of effort, ST2 will begin ESA processing on customer-supplied impeller blades, most likely from failed parts, or other factory rejects. In this task,

differences between the flat plate coating techniques and the curved impeller surfaces will be identified, and the process optimized as required.

Phase I Schedule



Phase I Technical results

Surface Studies

The electro-spark alloy process is a unique deposition process in which a metal or ceramic is deposited on another metal or ceramic via an electric arc, forming a nano-grained surface with full metallurgical bonding while inducing no heat affected zone (HAZ). Initial efforts are focused on determining the substrate conditions of the titanium base alloy for optimum adhesion, the potential need for a "butter layer" of a diffusion bond enhancement layer, and the thickness of the given alloy as a function of the coating strength, adhesion, and micro-cracking susceptibility.

To date, most emphasis has been placed on the formation of our 7473 alloy (tungsten carbide/tantalum carbide cobalt) on Ti-6-4. This alloy has the best database for previous wear/erosion applications. It has been developed with thickness layers of ~ 0.003 inches. Any build-up beyond that thickness is limited due to the poor electrical conductivity of the coating. Its initial contact is with the base titanium, however subsequent build-up requires that the arc be connected directly with the carbide layer, and this results in slow, spotty build up for additional thickness. Moreover, previous experience has determined that thicker layers build up internal stresses, and are more likely to chip and crack.

The following operating parameters were implemented in the production of the initial coatings:

Cover Gas:	Argon
Electrode Angle:	70 degrees
Electrode Geometry:	3/16 inch solid rod
Pulse Rate (Hz):	440
Capacitance (μ F):	20
Step rate:	280
Voltage output (V):	140-150
Current (A):	1-1.25
Velocity:	first pass ~ 0.3 inch/sec second pass ~ 0.4 inch/sec

Materials Tested:

- **ESA 7473 (WC/TaC/Co) on Ti-6Al-4V**
 - tungsten carbide
 - tantalum carbide
 - cobalt
- **ESA 7422 (WC/TiC/Co) on Ti-6Al-4V**
 - tungsten carbide
 - titanium carbide
 - cobalt
- **ESA 815 (Cr_3C_2 /15Ni) on Ti-6Al-4V**
 - chromium carbide
 - nickel
- **ESA 2274 (TiC/Ni-Mo) on Ti-6Al-4V**
 - titanium carbide
 - nickel
 - molybdenum
- **TiB₂ on Ti-6Al-4V**
 - titanium diboride
- **Ti-6-4 base alloy (uncoated)**

The various coatings were evaluated using an in-house ASTM particle erosion test fixture. In this test, ~ 50 micron alumina particles were passed through a high pressure nozzle at 500 ft/sec and allowed to impact the test article at both 30 and 90 degree impact angles. Weight loss measurements were made on the coated samples and the base titanium alloys, and a volumetric comparison was then calculated, based upon the difference in density between the coating and the base titanium alloy. Results of these first erosion test series (one minute duration) are shown in Figures 11 and 12 with a comparison overview in Figure 13.

Test: Solid Particle Erosion in Gas Stream

Test Conditions:

- Particulate: -50micron Al_2O_3 (ASTM Standard)
- Gas: Dry Air @ 68°F
- Particulate Velocity: 500 ft/sec
- Particulate Loading: 12 grams/minute
- Impingement Angle: 30° and 90°
- Nozzle: 0.180 inch diameter, 5.0 inch long
- Nozzle to sample distance: 0.500 inch
- Exposure time: 1.0 minute

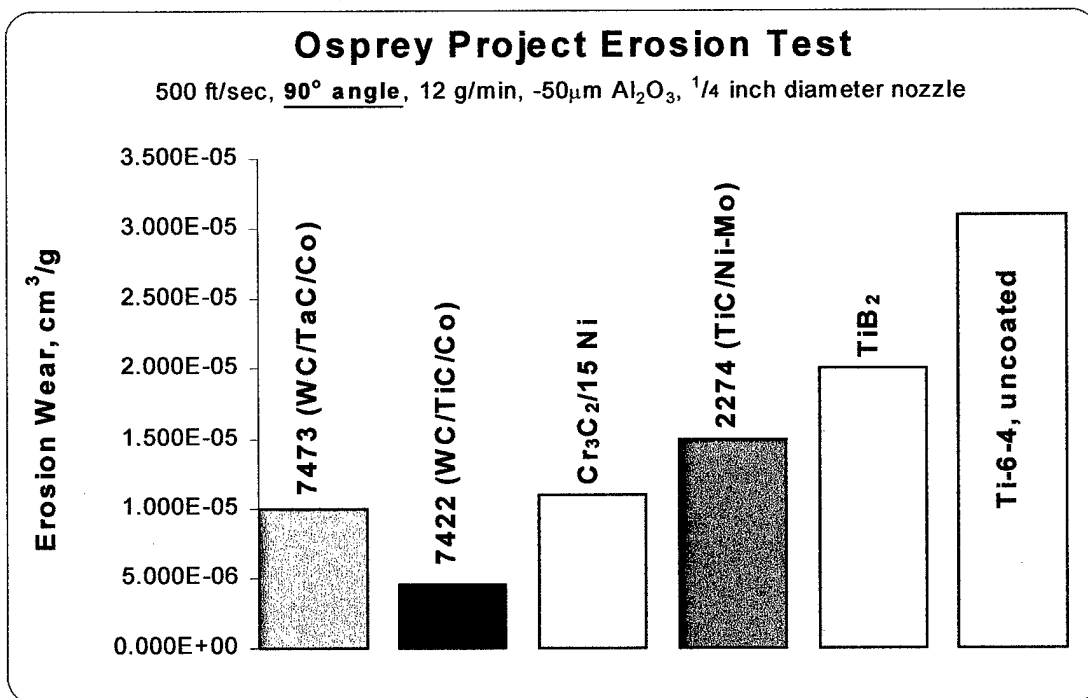


Figure 11. Results of the 1-minute erosion test at 90 degrees impact angle.

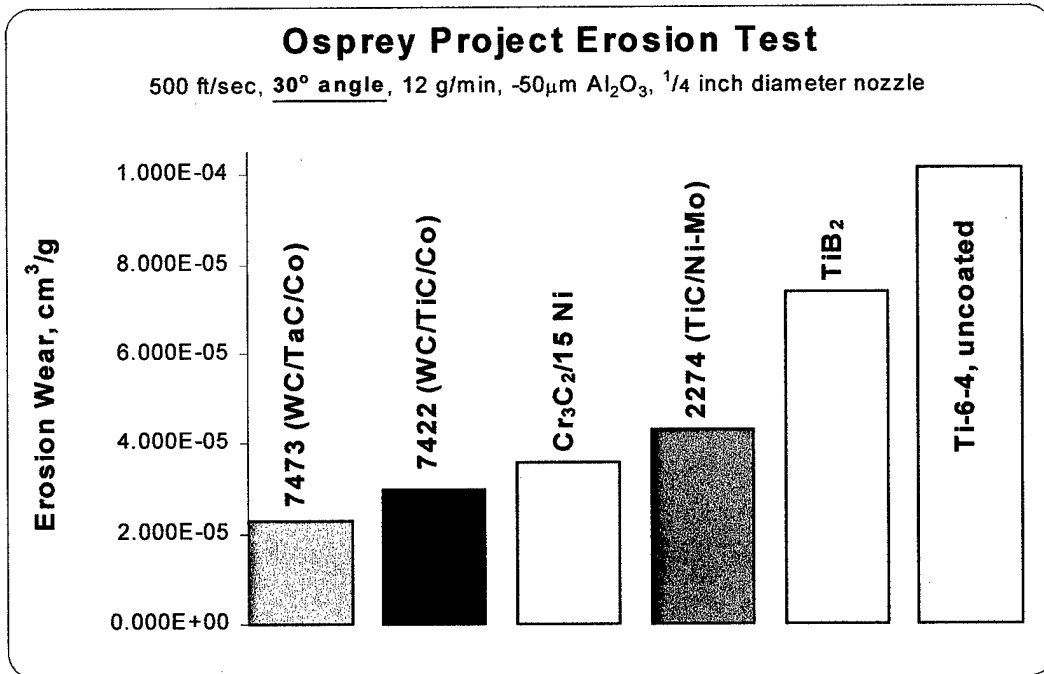


Figure 12. Results of the 1-minute erosion test at 30 degrees impact angle.

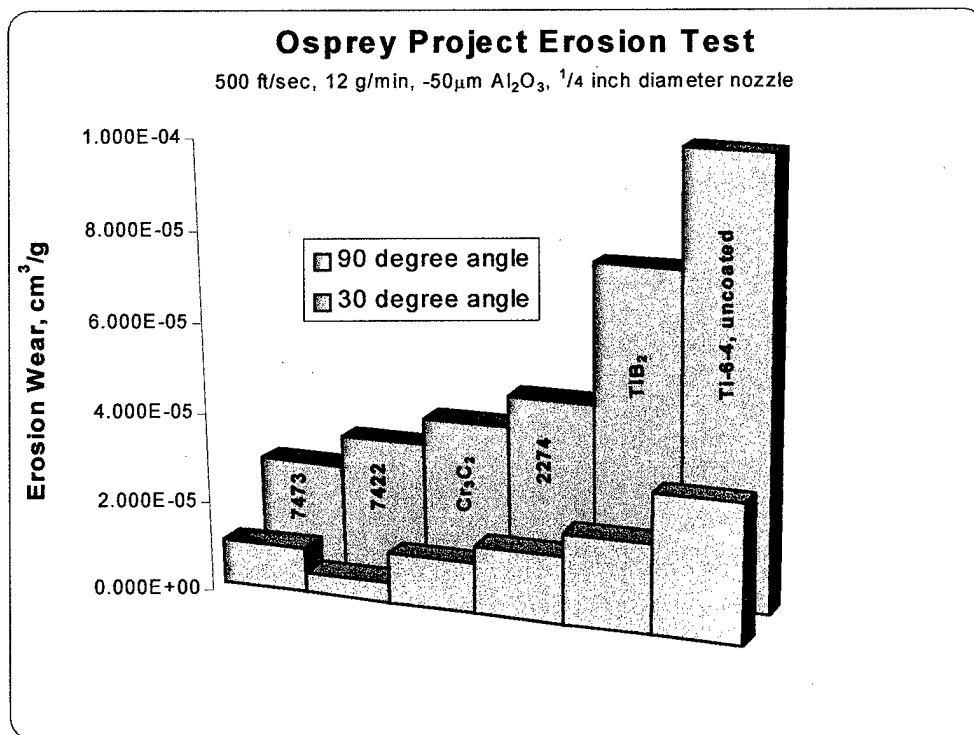


Figure 13. Comparison of the 30 and 90-degree impact data.

The data in Figure 13 shows a clear angular attack dependence on the coating's ability to protect the base alloy, with the 30-degree angle of attack being far more aggressive in erosion. It should be noted that, in all cases of erosion testing presented thus far, none of the coatings were breached.

In the second test series, the two best performers were evaluated under the same test conditions, but for a three-minute exposure. The results are presented in Figure 14. Based upon these results, the two best candidates: tungsten carbide/tantalum carbide/cobalt, and tungsten carbide/titanium carbide/tantalum carbide/cobalt were processed and provided to the University of Cincinnati for independent erosion testing.

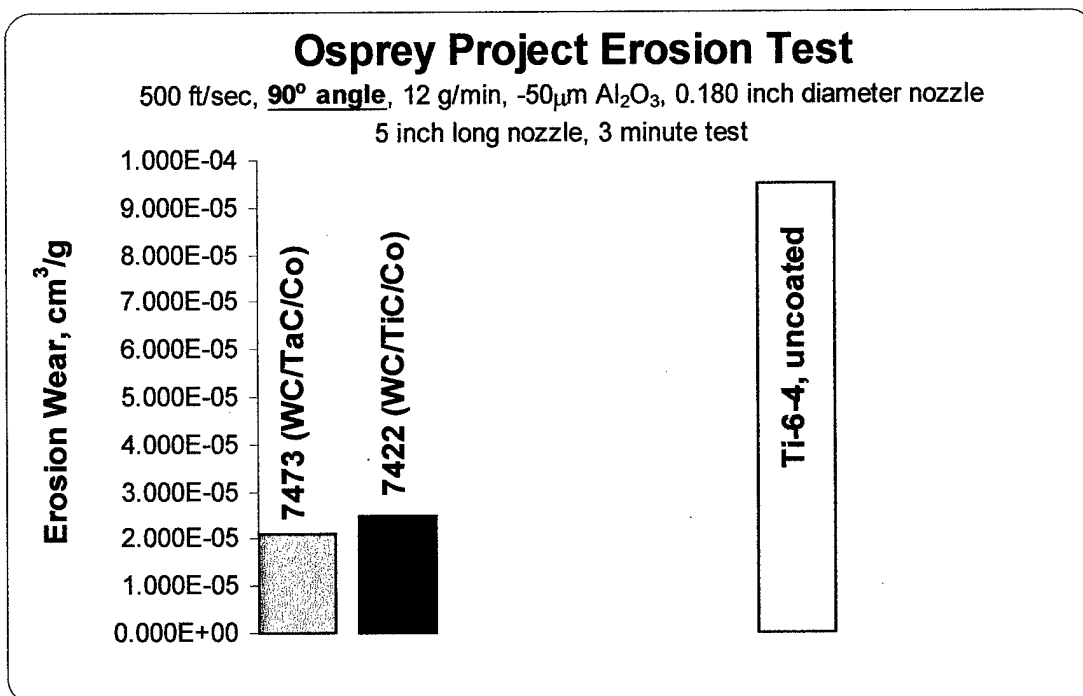


Figure 14. Three-minute erosion data, 90 degree angle, for base Ti-6-4, 7422, and 7473 electrode materials.

Wind Tunnel Testing – Series 1

After lengthy consideration, the NAVAIR customer has selected the wind tunnel test facility at the University of Cincinnati, under the direction of Professor Widen Tabakof, as the comparative test facility for the coatings from the 3 Phase I contractors under evaluation. While not much information has been gathered about the testing methodology employed at this facility, ST2 has submitted 2 initial test samples for erosion testing. These samples were coating 7473 (WC/TaC/cobalt) on Ti-6-4 with a coating thickness of ~ 0.002 inches and 7422

(WC/TiC/cobalt) on Ti-6-4 with the same thickness. Prof. Tabakof evaluated these coatings, and his initial findings indicate that wear was occurring too aggressively in his test fixture. The samples were ST2 for our own evaluation, and it was not clear what test methods were being employed by Prof. Tabakof. While one of our samples shows wear on the coating side, the second sample was tested on the bare Ti-6-4 side only. The conclusions made by Prof. Tabakof regarding our coatings are in error based upon our analysis.

The ST2 team re-evaluated our coating process, but decided that our coatings were performing adequately and that the University of Cincinnati was in error in its evaluation and determinations. Moreover, the University of Cincinnati provided direction to us on how to improve our coatings (make them thicker). While we acknowledge the University's expertise in erosion testing, we do not acknowledge their expertise on our coatings or our process, and will determine what changes if any, are necessary for our test coupons.

Wind Tunnel Testing- Series 2

In the second series of wind tunnel tests conducted by the University of Cincinnati, ST2 provided the identical coupon compositions as it did in the first test series. This time, it was acknowledged that our coatings performed well and were not breached. In a highly unusual test procedure, one coupon was tested using aluminum oxide grit, and was then inserted into a second series with 200-micron silica. The second test was compared directly to an untested coupon on bare Ti-6-4. Even under these conditions, the ST2 coupon performed better than the bare titanium, and again, the coating was not breached.

Despite the request by ST2 to obtain wear testing on both the 7473 and the 7422 coating chemistries in this second series of tests, the University of Cincinnati failed to accomplish this, and, instead, only provided additional data on the 7422 composition. This data continued to show the successful wear resistance trends already established by the 7422 composition.

Discussion of Results

As supplied, the raw data from the University of Cincinnati wind tunnel tests was subject to wide ranging interpretation. The ST2 team converted this data from weight loss into volumetric loss, since the density of the ST2 applied coatings were much higher than the density of the Titanium alloy. Using this approach, the wind tunnel Series 1 data is shown in Table 2 as provided by the University of Cincinnati. Then in Figures 15 and 16, the same data is presented in volumetric form. The numbers provided here correlate nicely to the volumetric wear data generated using our own in-house alumina grit-blast tests also conducted at 90 degrees (Figure 11). Although the impact particle loading is low

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for these tests, volumetric wear improvements ranged from 54X (alumina, 90 degrees) to 6.8X improvement realized by the coated titanium surfaces.

Alumina (9.5 microns) 90 degrees, 600 ft/sec:

(7422) WC/TiC/Co 0.156 mg/g

(7473) WC/TaC/Co 0.184 mg/g

Ti-6-4 base alloy 1.206 mg/g

Arizona Road Dust (1-100 microns) 90 degrees, 600 ft/sec:

(7422) WC/TiC/Co 2.3 mg/g

Ti-6-4 base 2.95 mg/g

Table 2. Univ. Cinn. Wind tunnel test data presented in weight loss numbers only, which do not take into account the density differences between the coatings and the base alloy

In the wind tunnel Round-2 tests, similar translation of the data from weight loss to volumetric loss supports the efficacy of the ESA-coatings. However, the test-team at the University of Cincinnati used a highly unusual test method in Round-2 that does not readily lend itself to comparison. Specifically, a coated sample was subjected to impact by 9.5-micron diameter alumina particles at 90 degrees at 600 ft/sec. Then, this same exposed sample was inserted into a test using 100-200 micron diameter silica particles also at 600 ft/sec. The data generated by this test was then compared directly to bare titanium. It must be noted that under typical test conditions, the bare titanium would only have been compared to a pristine coating, rather than one that has already undergone some degree of wear. As long as this test was repeated identically to samples supplied by other vendors, then (and only then) can the data be considered valid for the purposes of comparison.

It should also be noted that, in this unusual test approach, volumetric calculations indicate that the ESA coated surface had still not been breached.

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vendors, then (and only then) can the data be considered valid for the purposes of comparison.

It should also be noted that, in this unusual test approach, volumetric calculations indicate that the ESA coated surface had still not been breached, as shown in the comparative micrographs in Figure 17.

Throughout the wind tunnel test series, several irregularities were noted in the test procedures and data provided by the University of Cincinnati. ST2 feels that these irregularities may have a bearing on the conclusions made by the customer, and therefore enumerates below the specific issues raised during the test series:

1. Univ. of Cinn. personnel did not adequately track the sample IDs placed on the coupons by ST2, nor did they offer a ready cross-reference with their own identification numbers
2. Univ. of Cinn. personnel provided ST2 data in Series 1 in which a sample was placed into test backwards, exposing bare titanium to the erosion, and the data was presented as that of a coated sample
3. Univ. of Cinn. personnel attempted to guide ST2 in how to improve their coatings with no knowledge of the ST2 process. Their suggestions, if followed, would have only made ST2 performance worse, and their suggestions were based upon an incorrect interpretation of the data generated.
4. Univ. of Cinn. presented ST2 data and hardware to the Navy customer, indicating that the ST2 coatings were not performing. ST2 was not given the data by the Univ. of Cinn, but rather had to request it from the COTR. Only then, were ST2 personnel able to correctly show that the Univ. of Cinn. data was incorrectly interpreted, and offer the NAVAIR customer insight into the correct interpretation of the data
5. In Series 2, the Univ. of Can, failed to test both chemistries provided by ST2 for evaluation. With 5 days left in the contract, this data was finally made available to ST2. At that point, ST2 requested an immediate test of the second chemistry in order to present as much data as possible in the final report. The Univ. of Cinn. agreed to perform the tests immediately. However, the next day, ST2 received data from the same chemistry, and not the data that it had requested on the second coating chemistry provided. This occurred at the end of a week, only permitting one day before the tests could be conducted and entered into the final report. Therefore, this data cannot be entered into the report at this time.

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Tabakof data (dated Aug 16, 2000)

Materials Tested:

- ESA 7473 (WC/TaC/Co) on Ti-6Al-4V
- ESA 7422 (WC/TiC/TaC/Co) on Ti-6Al-4V
- Ti-6Al-4V (uncoated)

Test Conditions:

- Erodent powder: -9.5mm Al₂O₃ and Arizona dust powder (silica, 1-100 μm)
- Ambient temperature (approximately 70°F)
- Particulate Velocity: 600 ft/sec
- Exposure time:
- Particles weight impacting the tested sample: 5 or 10 grams
- Angle of particle impact: 30° and 90°

ESA Coating	specific gravity, g/cm ³	erosion rate (per Tabakov's data), g/g for 1 inch square area	loss, cm ³ /g X 10 ⁻⁵
600 ft/sec, 90° angle, -9.5mm Al ₂ O ₃			
7473 (WC/TaC/Co)	13.86	0.002300	16.59
7422 (WC/TiC/Co)	12.64	0.000170	1.34
Ti-6-4, uncoated	4.51	0.002950	65.41
600 ft/sec, 30° angle, Arizona dust powder 1-100μm			
7473 (WC/TaC/Co)	13.86	n/a	n/a
7422 (WC/TiC/Co)	12.64	0.000492	3.89
Ti-6-4, uncoated	4.51	0.001206	26.74

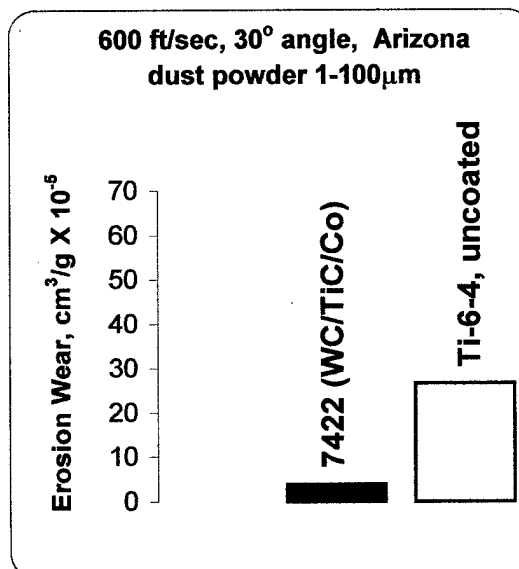
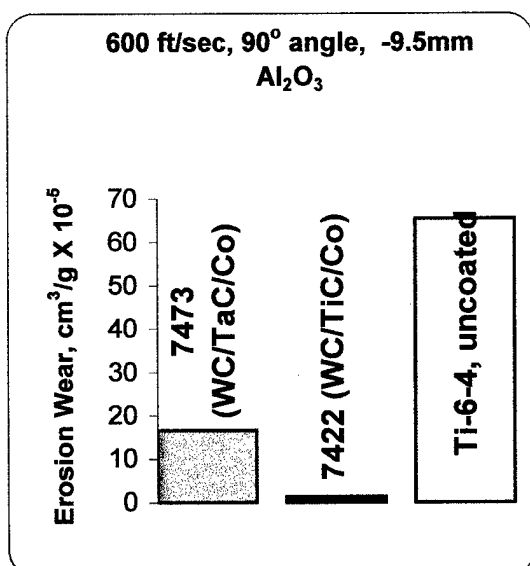


Figure 15. Volume-specific analysis of wear data from Round-1 of wind tunnel testing at the University of Cincinnati Wind Tunnel test facility.

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Tabkof's data (dated Oct 3, 2000)

Materials Tested:

- ESA 7422 (WC/TiC/TaC/Co) on Ti-6Al-4V
- Ti-6Al-4V (uncoated)

Test Conditions:

- Erodent powder: -9.5mm Al₂O₃ and Arizona dust powder (silica, 1-100 mm)
- Particulate Velocity: 600 ft/sec
- Particles weight impacting the tested sample: 50, 60 or 100 grams
- Angle of particle impact: 30° and 90°

ESA Coating	specific gravity, g/cm ³	erosion rate (per Tabakof's data), g/g for 1 inch square area	loss, cm ³ /g X 10 ⁻⁵
600 ft/sec, 90° angle, -9.5mm Al ₂ O ₃			
7422 (WC/TiC/Co)	12.64	0.000200	1.58
Ti-6-4, uncoated	4.51	n/a	n/a
600 ft/sec, 30° angle, -9.5mm Al ₂ O ₃			
7422 (WC/TiC/Co)	12.64	0.000794	6.28
Ti-6-4, uncoated	4.51	n/a	n/a

600 ft/sec, 90° angle, (Silica, 100-200 mm)			
7422 (WC/TiC/Co) <i>After tested with - 9.5mm Al₂O₃</i>	12.64	0.001802	14.26
Ti-6-4, uncoated	4.51	0.001563	34.66

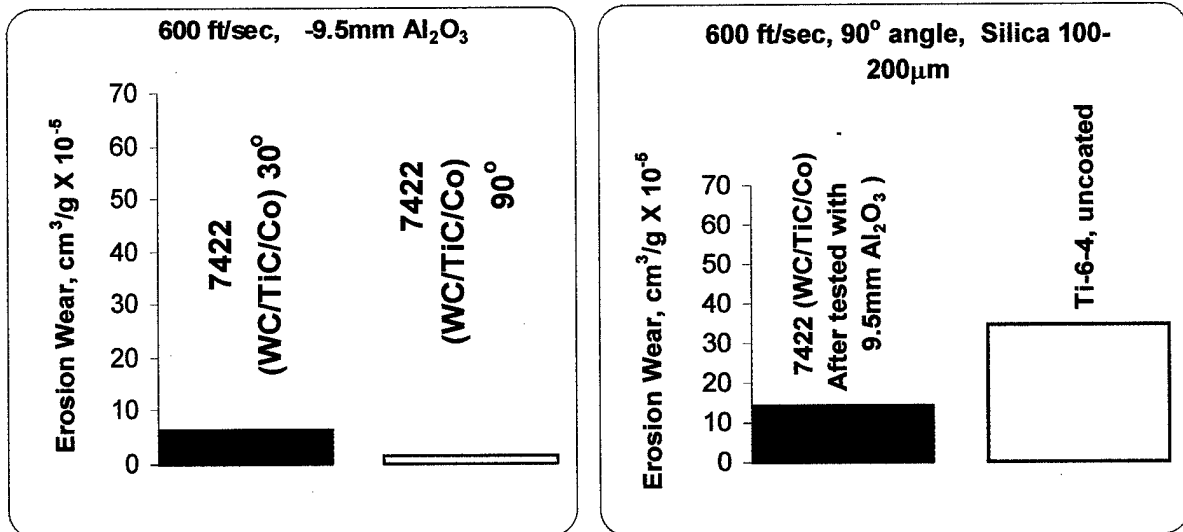


Figure 16. Volume-specific analysis of wear data from Round-2 of wind tunnel testing at the University of Cincinnati Wind Tunnel test facility.

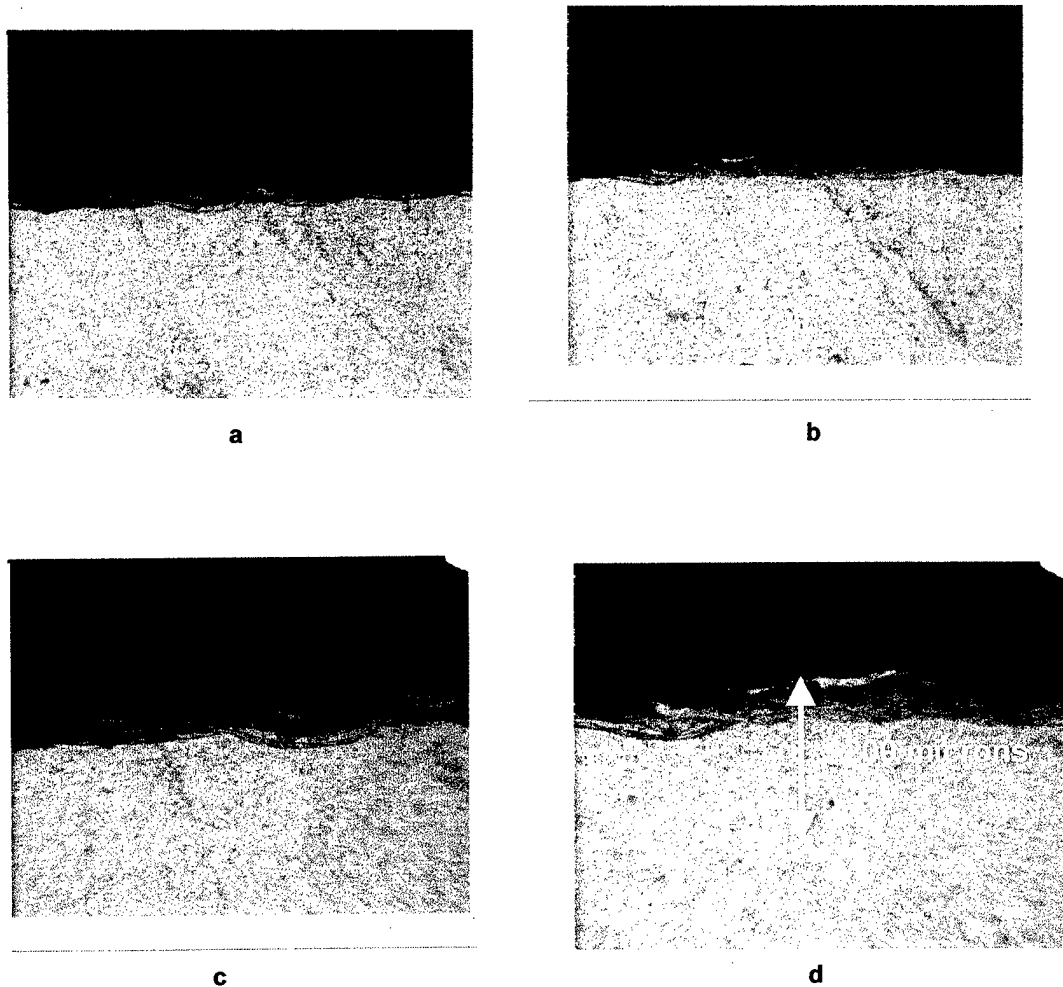


Figure 17. Polished cross-section of Ti-6-4 coated with tungsten carbide/tantalum carbide cobalt after wind tunnel erosion testing (Series-1). Note the presence of the coating in the impact area as compared to a shielded portion of the coupon where no erosion occurred. (a-b) 100X with un-tested 7422 coating on left and erosion test sample on right. (c-d) 200X comparison on untested coating (right) and erosion tested coating (left). Note the similarities between the tested and the untested surfaces, indicating that little, if any wear has taken place under these test conditions.

Manufacturability Study

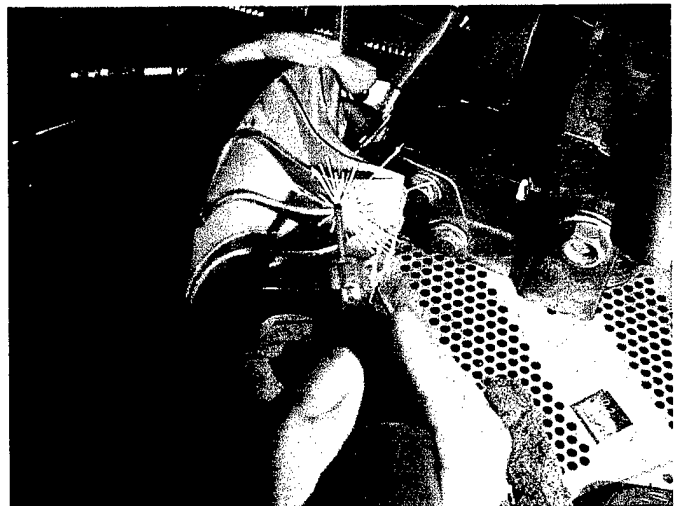
In addition to the Phase I test series that have been conducted on the erosion properties of the ST2 coatings, a detailed study was also conducted on the ability to provide the ESA coatings in a repeatable, affordable manner for OEM production needs. Phase I studies did not afford the ability to develop processing algorithms to program robotics and streamline the coating process for complete automation, however, we believe that the ESA coating process does lend itself to full automation for this intricate hardware item. It should be stressed here that the ESA process is achieved through physical contact of an electrode on the part being coated. For this geometry, it is possible to develop a robotic program that coats the appropriate areas on a single impeller vane, then simply index the impeller to the next vane and repeat the process, offering a high degree of repeatability and reliability to the process.

In this effort, a worn impeller was obtained from the manufacturer, Honeywell. This part was inspected and then ESA coatings were applied to the hardware, using manual operations, to demonstrate the ease with which the intricate blades could be coated. The following series of photos (Figure 18) show the coating process and the resultant hardware from that process. Under separate cover, the coated blade will be provided to the government contact for demonstration purposes only. The blade was severely damaged under tests conducted prior to our receipt, and is in no way intended to be evaluated in any erosion test hardware.

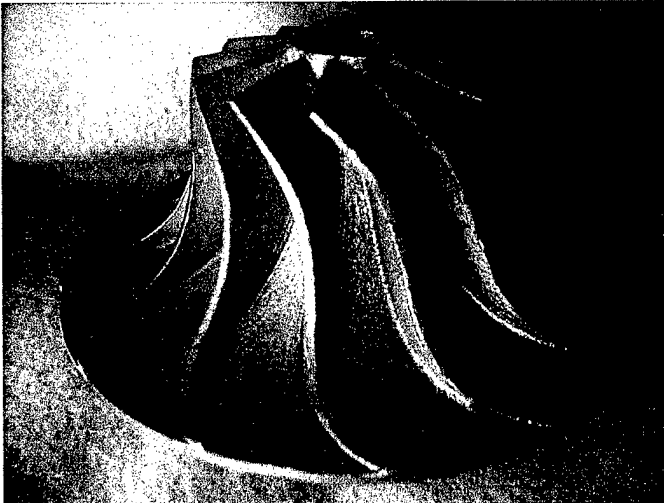
Figure 18. Photo essay on procedures for the manual coating of the impeller blades.



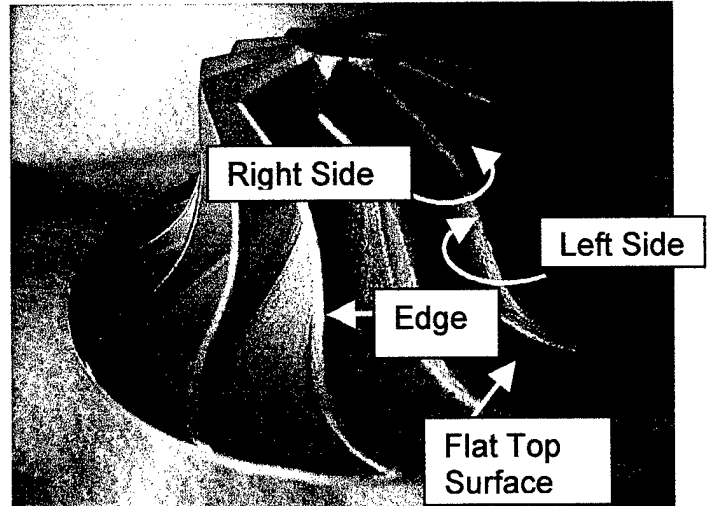
Grinding the "contaminated" surface



ESA applied to the surfaces



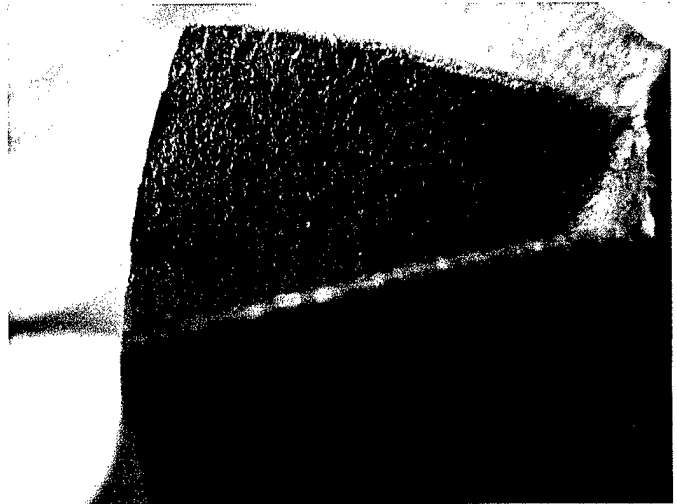
The Impeller with four segments coated.



Nomenclature for the Impeller Surfaces



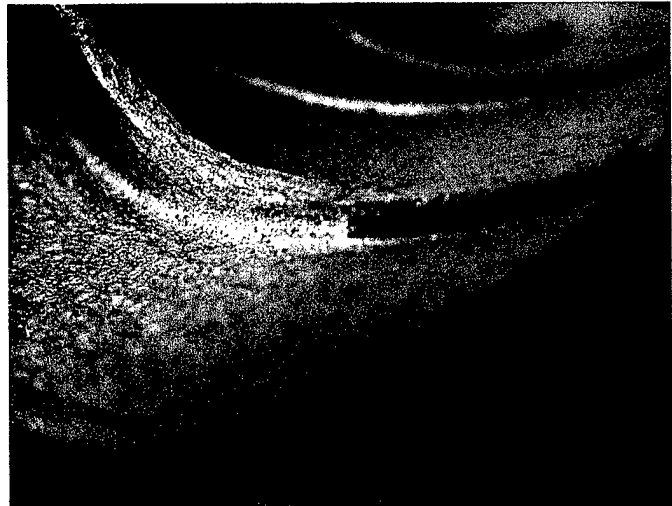
The inside corners coated



The ESA surface texture



The deformed edge



Coating on the deformed edge

Note that, in this manual coating demonstration, a surface texture is visible in the photos provided. While ESA does produce a slight texture, it can be buffed to reduce this roughness, however, the overall texture is highly uniform and it is not believed will effect the balance requirements on this high-RPM component. Due to the damage already existing on the part that was provided, certain geometric capabilities of the ESA process could not be adequately demonstrated. However, the overall hardware geometry will provide no major issues to achieve a uniform ESA coating over the major wear areas of concern.

It is estimated that, on the 13-blade impeller used on the V-22, that there is ~ 4 square inches of coating required per blade, of 52 square inches per impeller. An automated ESA approach should be sufficient to deliver this coating in a cost effective manner.

Recommendations

From our own wear testing data, as well as from that generated independently by the University of Cincinnati; coatings deposited by the ESA process demonstrated a significant improvement over the base titanium alloys. There is still sufficient uncertainty arising from the test methodology employed in the wind tunnel testing that we are unable to independently validate the accuracy of the data generated at that facility. In some cases, there is correlation with our own wear data, however, test-to-test consistency was not well controlled, and

quantitative numbers may be difficult to corroborate. ST2 is concerned that this unreliability may also be an issue across the board for the other coatings tested in this effort, and that this fact alone may nullify some of the data generated.

The fact that, after all impact tests are concluded, the ESA coatings were able to survive and offer wear improvement numbers as high as 6X for large particle impact testing, the value of this process warrants further investigation.

In the optional Phase I task, ST2 recommends the following specific testing:

- identically prepared base alloy coupons be supplied to one or all competitive vendors for coating
- that each coated test sample be evaluated at the Univ. of Cincinnati test facility
- that each competitor, together with the Navy COTR, be present during the testing of the samples, and be allowed to scrutinize the test plan prior to, and during the test cycle
- that each vendor assist in insuring the accuracy of the test protocol (data recording, sample ID. Etc.) since these steps were suspect in the initial testing

Should the data generated to date be sufficient to make a down-selection for one or more Phase II candidates, that all three candidates be convened for a data review meeting in which the results of the competition are made available for discussion among all 3 parties.

If, after this optional test series is conducted there is still an issue with the quality of the data being produced by the Univ. of Cinn. Facility, then ST2 strongly recommends that a new facility be identified where more reliable, repeatable test procedures can be achieved.

Interim Task Outline

ST2 suggests the following specific tasks for its own Optional Task effort:

1. Prepare additional test coupons for wind tunnel erosion testing
2. Perform additional ASTM erosion tests in our facility, using identical grit and velocity conditions as those to be used in the wind tunnel
3. Have ST2 personnel present to witness any wind tunnel tests that are conducted
4. Take part in any round-robin data analysis conducted by NAVAIR with the other competitors in this effort

Phase II Outline

A full Phase II proposal will be delivered to the customer upon their recommendation. It will contain the following key elements:

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- deliver test coupons for new wind tunnel (or other) erosion tests that better mimic the real-world conditions being experienced by the impeller hardware on the V-22
- optimize the ESA process for robotic control on the impeller hardware design
- perform full coatings tests of new impeller hardware using an automated ESA system
- deliver documentation of hardware coating parameters, robotic requirements, QA, etc. for transition into Phase III production hardware for the customer (to be applied either through a license to the manufacturer, or to be applied by ST2 via a job-shop production contract)
- assess additional/similar wear and erosion needs on existing hardware systems and attempt to exploit the capabilities developed under this Phase II program for additional military as well as commercial exploitation

Based upon the efforts conducted in Phase I, the ST2 team is convinced that it has developed and evaluated coatings that are capable of extending the life of the V-22 impeller sufficiently to meet customer life-cycle maintenance goals. The process developed is highly repeatable, able to produce a consistently reliable coating, and able to meet automation production capabilities that will meet affordability factors that may exist.